CANDIDATE NAME	( )	CLASS	17S0		

RAFFLES INSTITUTION
2017 YEAR 6 PRELIMINARY EXAMINATION

Higher 2

CHEMISTRY 9729/04

Paper 4 Practical

17 August 2017

2 hours 30 minutes

Do NOT turn over the Question Booklet until you are told to do so.

READ THESE INSTRUCTIONS FIRST.

Write your name and class on the space provided when instructed to do so.
Give details of the practical shift and laboratory where appropriate, in the space provided.

Write in dark blue or black pen.
You may use a HB pencil for any diagrams or graphs.
Do not use staples, paper clips, glue or correction fluid.

Answer all questions in the spaces provided on the Question Paper.
The number of marks is given in brackets [ ] at the end of each question or part question.

The use of an approved scientific calculator is expected, where appropriate.
You may lose marks if you do not show your working or if you do not use appropriate units.
Qualitative Analysis Notes are printed on pages 17 and 18.

Shift	
Laboratory	
Bench Number	

For Examiner's Use	
Question	**Marks**
1	**/ 24**
2	**/ 16**
3	**/ 15**
Total	**/ 55**

This Question Paper consists of **18** printed pages.

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1 Determination of *x* in hydrated copper(II) sulfate, $CuSO_4 \bullet xH_2O$ by titrimetric analysis

The formula of hydrated copper(II) sulfate is $CuSO_4 \bullet xH_2O$, where ***x*** represents the number of moles of water of crystallisation in one mole of the compound.

You are to determine the value of ***x*** by titrating a solution of iodine, I_2, with a solution of thiosulfate ions, $S_2O_3^{2-}$. The iodine is formed by the reaction of copper(II) ions, Cu^{2+}, with iodide ions, I^-.

$$2Cu^{2+} + 4I^- \rightarrow 2CuI + I_2$$
$$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$$

You are provided with the following.

FA 1 is hydrated copper(II) sulfate, $CuSO_4 \bullet xH_2O$.
FA 2 is 0.050 mol dm^{-3} sodium thiosulfate, $Na_2S_2O_3$.
FA 3 is 1.0 mol dm^{-3} potassium iodide, KI.

(a) Preparation of aqueous copper(II) sulfate, FA 4

1. Weigh accurately about 3.1 g of **FA 1** in a clean and dry weighing bottle. Record the mass used in an appropriate format in the space provided on page **3**.

2. Transfer the **FA 1** quantitatively into a 100 cm^3 beaker and dissolve it in some deionised water. Rinse the weighing bottle with deionised water several times, adding each rinsing to the beaker. Using a glass rod, stir the solution in the beaker until all the **FA 1** dissolves.

3. Transfer the solution and washings from the beaker to a 250 cm^3 volumetric flask.

4. Make up the solution to the 250 cm^3 mark of the flask with deionised water, then stopper and mix thoroughly by inverting the flask a number of times.

5. Label the solution **FA 4**.

(b) (i) Titration of iodine against FA 2

1. Fill the burette with **FA 2**.

2. Using a pipette, transfer 25.0 cm^3 of **FA 4** into a conical flask.

3. Use a measuring cylinder to add approximately 7 cm^3 of **FA 3** into the same conical flask. This will form a brown mixture consisting of iodine and an off-white precipitate.

4. Titrate this mixture with **FA 2** until most of the brown has been removed, and then add approximately 5 cm^3 of starch indicator. The mixture will turn a blue-black colour.

5. Continue titrating this mixture until the blue-black colour just disappears to leave behind the off-white precipitate. This is the end-point of the titration.

6. Repeat the titration as many times as necessary to provide consistent results.

7. Record titration results in the space provided below. Recorded results must show the precision of your practical work.

Results

[7]

(ii) From your titration results, obtain a suitable volume of **FA 2** to be used in your calculations. Show clearly how you obtained this volume.

volume of **FA 2** = …….....[1]

(c) (i) Calculate the amount, in moles, of thiosulfate ions that were present in the volume of **FA 2** calculated in **(b)(ii)**.

amount of $S_2O_3^{2-}$ = …….....[1]

(ii) Use your answer to **(c)(i)** to obtain the amount, in moles, of copper(II) ions present in 25.0 cm^3 of **FA 4**.

amount of Cu^{2+} in 25.0 cm^3 of **FA 4** = …….....[1]

(iii) Determine the concentration, in mol dm^{-3}, of copper(II) ions in **FA 4**.

concentration of Cu^{2+} in **FA 4** = …….....[1]

(iv) Use your answer to **(c)(iii)** to calculate the M_r of $CuSO_4 \bullet \boldsymbol{x} H_2O$.

M_r of $CuSO_4 \bullet \boldsymbol{x} H_2O$ = …….....

Hence, deduce the value of ***x***. Show your working.
[A_r: Cu, 63.5; O, 16.0; H, 1.0; S, 32.1]

x = …….....[3]

(d) The maximum error in a single burette reading is ± 0.05 cm^3.

When titrating iodine against **FA 2**, a student recorded that 23.40 cm^3 of **FA 2** was used. However, the actual volume of **FA 2** added was 23.50 cm^3.

Explain how the error may have arisen from using a burette to obtain an individual titre volume.

……..

…….....[1]

(e) Planning

When aqueous solutions of copper(II) sulfate and sodium carbonate are combined, a solid known as basic copper(II) carbonate, $Cu(OH)_2 \bullet nCuCO_3$, precipitates from solution.

$Cu(OH)_2 \bullet nCuCO_3$ decomposes to copper(II) oxide when heated by a Bunsen flame.

The percentage by mass of copper in this basic copper(II) carbonate can be determined in the laboratory using a gravimetric technique.

(i) Write a chemical equation for the thermal decomposition of $Cu(OH)_2 \bullet nCuCO_3$.

……...[1]

(ii) Outline, step by step, the practical sequence for the method you would use to

- carry out the weighing of a sample of $Cu(OH)_2 \bullet nCuCO_3$ solid,
- decompose this sample by heating, and
- ensure that the decomposition is complete.

You may assume that the following are available:

- 5 g of $Cu(OH)_2 \bullet nCuCO_3$
- crucible
- pipe-clay triangle
- weighing balance
- Bunsen burner
- all other common lab apparatus

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………[4]

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(iii) Prepare a table in the space below to show the masses you would measure and record during the experiment. Include in your table any other masses you would calculate from the experimental results to enable you to determine the percentage by mass of copper in the mixture. Insert in your table the letters **A**, **B**, **C**, etc. to represent each mass.

[2]

(iv) Use the letters you have written in **(e)(iii)** to show how you would process the results to find the percentage by mass of copper in $Cu(OH)_2 \bullet nCuCO_3$.
[A_r: Cu, 63.5; O, 16.0; C, 12.0; H, 1.0]

[2]

[Total: 24]

2 The reaction between aqueous potassium peroxodisulfate, $K_2S_2O_8$, and aqueous potassium iodide, KI, produces iodine as a product.

$$K_2S_2O_8(aq) + 2KI(aq) \longrightarrow I_2(aq) + 2K_2SO_4(aq) \quad \textbf{\textit{reaction A}}$$

The rate of this reaction will be monitored by visual colorimetry.

You are provided with the following.

FA 3 is 1.0 mol dm^{-3} potassium iodide, KI, as used in Question **1**.
FA 5 is 0.020 mol dm^{-3} potassium peroxodisulfate, $K_2S_2O_8$.
FA 6 is 0.020 mol dm^{-3} iodine, I_2.

(a) Preparation of iodine solutions of known concentrations

You are to prepare iodine solutions of different concentrations as shown in the table below, by diluting appropriate volumes of **FA 6** to a total volume of 40 cm^3.

$[I_2]$ / mol dm^{-3}	**Volume of FA 6 used / cm^3**	**Total volume of mixture / cm^3**
0.001	2.00	40
0.002	4.00	40
0.004	8.00	40
0.006	12.00	40
0.008	16.00	40

1. Fill a burette with **FA 6**.

2. Transfer the required volume of **FA 6** into a 50 cm^3 measuring cylinder.

3. Add deionised water into the same measuring cylinder until the 40 cm^3 mark is reached.

4. Transfer the solution into a clean and dry 100 cm^3 beaker labelled with its concentration.

5. Repeat steps **1** to **4** to prepare iodine solutions of the other concentrations.

6. Arrange the beakers in order of increasing concentration on the blank piece of A4 paper provided.

These five beakers contain your reference solutions to be used in **(b)**.
The colour in each beaker corresponds to a particular $[I_2]$ as listed in the table above.

(b) Monitoring the rate of reaction between potassium peroxodisulfate and potassium iodide

As the intensity of the yellow colour of iodine is proportional to its concentration, the rate of ***reaction A*** can be monitored by comparing the colour of the reaction mixture to samples of aqueous iodine of known concentrations.

For this experiment, the times taken to reach the different concentrations of iodine matching those prepared in **(a)** are to be recorded.

In this investigation, potassium iodide is used in large excess over potassium peroxodisulfate.

You are strongly advised to read through the entire experimental procedure below, from steps **1** to **6**, before starting the experiment.

1. Transfer 20 cm^3 of **FA 5** into a clean and dry 100 cm^3 beaker using a 25 cm^3 measuring cylinder.

2. Measure 20 cm^3 of **FA 3** using a separate 25 cm^3 measuring cylinder.

3. Transfer the sample of **FA 3** into the beaker containing **FA 5** and start the stopwatch. Swirl the flask once to ensure thorough mixing before leaving it to stand.

4. While keeping the stopwatch running, record the time taken, **to the nearest second**, for the concentration of iodine produced in the reaction mixture to reach 0.001 mol dm^{-3} i.e. for the colour of the reaction mixture to **match** the colour of the reference solution containing 0.001 mol dm^{-3} of aqueous iodine which you have prepared in **(a)**.

You should observe the colour of the solutions by viewing the beakers from **above**. **Do not** place your face too close to the beakers.

5. Continue to record the time taken for the concentration of iodine produced in the reaction mixture to reach the concentrations of the remaining samples prepared in **(a)**.

The time taken for the concentration of iodine produced in the reaction mixture to reach 0.008 mol dm^{-3} is more than 120 s.

6. Tabulate your results in a suitable table in the space below.

(i) Results

[2]

(ii) One reason potassium iodide was added in large excess over potassium peroxodisulfate was to ensure that the peroxodisulfate ions are completely reacted in the reaction mixture.

Suggest another reason to explain why this was necessary.

……..

……..

…….....[1]

(iii) Calculate the concentration of $S_2O_8^{2-}$ in the reaction mixture immediately after **FA 3** and **FA 5** were mixed.

concentration of $S_2O_8^{2-}$ immediately after mixing = …….....[1]

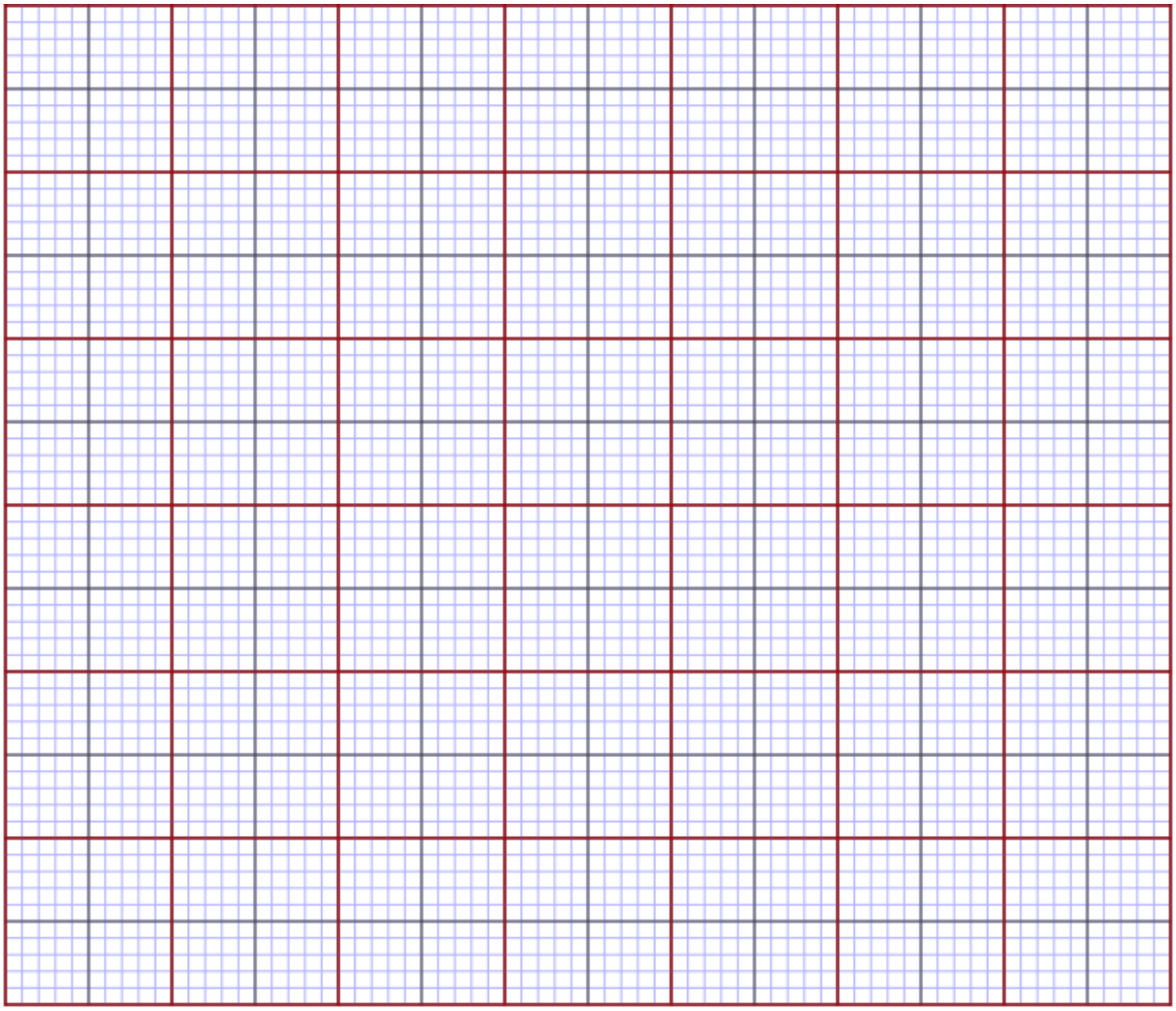
(iv) Using your answer to **(b)(iii)**, calculate the concentration of $S_2O_8^{2-}$ remaining in the reaction mixture when the concentration of iodine produced was 0.001 mol dm^{-3}.

concentration of $S_2O_8^{2-}$ remaining in reaction mixture = …….....[1]

(v) Hence, record, on your table in **(b)(i),** the concentrations of $S_2O_8^{2-}$ remaining in the reaction mixture when 0.001, 0.002, 0.004, 0.006 and 0.008 mol dm^{-3} of iodine were produced.

[1]

(c) Using data from **(b)(i)**, on the grid below, plot a graph of the concentration of potassium peroxodisulfate on the *y-axis* against time on the *x-axis*. You should draw the line of best fit.



[3]

(d) The order of reaction with respect to potassium peroxodisulfate is 0, 1 or 2.

Use your graph to determine the order of reaction with respect to potassium peroxodisulfate. Clearly show all construction lines and working.

order of reaction with respect to $K_2S_2O_8$ = …….....[3]

(e) Further experiments were carried out by varying the concentrations of potassium iodide and potassium peroxodisulfate.

Initial [KI] / mol dm^{-3}	Initial [$K_2S_2O_8$] / mol dm^{-3}	Initial rate / mol dm^{-3} s^{-1}
0.10	0.020	9.10×10^{-4}
0.20	0.020	3.63×10^{-3}

(i) Determine the order of reaction with respect to KI.

order of reaction with respect to KI = …….....[1]

(ii) Hence write down the rate equation for the reaction between potassium iodide and potassium peroxodisulfate.

…….....[1]

(f) The uncertainty (error) associated with **each reading** using a 25 cm^3 measuring cylinder is $\pm$0.5 cm^3.

Calculate the maximum total percentage uncertainty (error) in the **volume** of the **reaction mixture** in **(b)**.

maximum total percentage uncertainty (error) = …….....[1]

(g) In **(a)**, a student accidentally added deionised water into the measuring cylinder until the 50 cm^3 mark, instead of the 40 cm^3 mark, for all his reference solutions.

Explain the effect of this mistake on the times recorded in **(b)(i)**.

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…….....[1]

[Total: 16]

3 Inorganic and Organic Analysis

(a) You are provided with **FA 7** and **FA 8**.

FA 7 is an aqueous solution containing one cation and one anion.
FA 8 is a reducing agent.

You are to perform a series of test-tube reactions described in **Table 3.1**, and record your observations in the table. Use the observations to help you identify the cation and anion in **FA 7**, and to deduce the identity of **FA 8**.

In all tests, the reagent should be added gradually until no further change is observed unless you are instructed otherwise.

You should indicate clearly at which stage in a test a change occurs, recording your observations alongside the relevant tests. Your answers should include

- details of colour changes and precipitates formed,
- the identities of gases evolved and details of the test used to identify each gas.

Table 3.1

	Test	Observations
(i)	To 1 cm depth of **FA 7** in a test-tube, add an equal depth of aqueous sodium carbonate.	
(ii)	To 1 cm depth of **FA 7** in a test-tube, add 1 cm depth of dilute hydrochloric acid, then	
	add 1 cm depth of aqueous barium chloride.	

Table 3.1

	Test	Observations
(iii)	To 2 cm depth of dilute sulfuric acid in a boiling tube, add 1 spatula of **FA 8**. Warm the mixture **cautiously** for one minute. Leave the mixture to cool and decant the solution into another test tube. To 1 cm depth of the resulting solution, add dilute aqueous ammonia.	
(iv)	To 3 cm depth of **FA 7** in a boiling tube, add 2 spatulas of **FA 8**. Warm the mixture **cautiously** for one minute, then leave to cool and filter the mixture. To 1 cm depth of the filtrate, add dilute aqueous sodium hydroxide.	

[8]

(v) Complete **Tables 3.2** and **3.3** below, using the observations in **Table 3.1** to identify the compounds **FA 7** and **FA 8**.

In each case, give evidence to support your conclusion.

Table 3.2

Ions present		**Evidence**
FA 7		

[2]

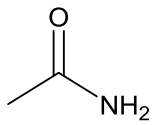
Table 3.3

Identity of FA 8	**Evidence**

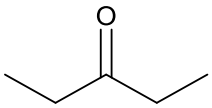
[1]

(b) Planning

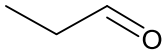
Consider the following organic compounds.



ethanamide



pentan-3-one



propanal

You are provided with aqueous solutions of the three organic compounds above.

Plan an investigation, using test-tube reactions, which would allow you to identify each of these three organic compounds.

Each compound should be identified by at least one positive test result. It is not sufficient to identify a compound simply by eliminating all the others.

Your plan should include:

- details of the reagents and conditions to be used,
- an outline of the sequence of steps you would follow,
- an explanation of how you would analyse your results in order to identify each compound.

Once a compound has been clearly identified, your plan should concentrate on distinguishing the remaining compounds.

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……..[4]

[Total: 15]